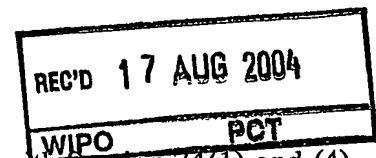




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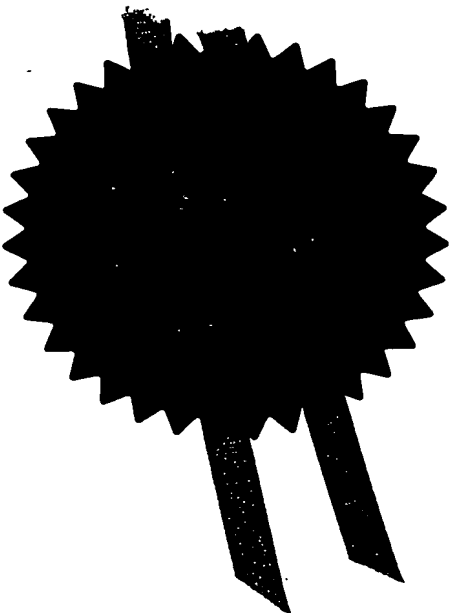


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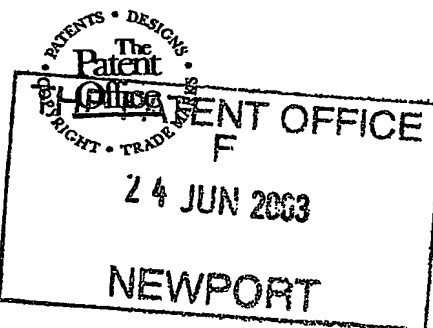


Signed 

Dated 5 July 2004

# Patents Form 1/77

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24JUN03 EB17391-3 D00350  
P01/7700 0.00-0314671.9

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The Patent Office

Cardiff Road  
Newport  
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1. Your reference

MG/LW/P/23965.GB

2. Patent application number

(The Patent Office will fill in this part)

0314671.9

24 JUN 2003

3. Full name, address and postcode of the or of each applicant (underline all surnames)

W. & J. LEIGH & CO.,  
TOWER WORKS,  
KESTOR STREET,  
BOLTON, LANCASHIRE.  
BL2 2AL.

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

8159204001  
UNITED KINGDOM.

4. Title of the invention

INTUMESCENT COATING COMPOSITIONS

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

WILSON GUNN M'CAW  
41-51 ROYAL EXCHANGE  
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MANCHESTER  
M2 7BD

Patents ADP number (if you know it)

7153927001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number  
(if you know it)

Date of filing  
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7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing  
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8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if

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- a) any applicant named in part 3 is not an inventor, or
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## Patents Form 1/77

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Description

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Claim(s)

Abstract

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Priority documents

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Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11. I/We request the grant of a patent on the basis of this application.

Signature

Date 23/6/2003

Wilson Gunn McGraw.

12. Name and daytime telephone number of person to contact in the United Kingdom MR. MARK GOODWIN 0161-827-9400

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## INTUMESCENT COATING COMPOSITIONS

The present invention relates to liquid intumescent coating compositions that have particular, but not exclusive, application in protecting steel structures in  
5 a fire situation.

Intumescent coating compositions are commonly used to protect structural steel components in buildings against the effects of cellulosic fire conditions. They contain a resin system "pigmented" with various intumescent ingredients that under the influence of heat, react together to produce an insulating foam or "char",  
10 having low thermal conductivity, which has a volume many times that of the original coating. This char greatly reduces the rate of heating experienced by the steel, thus extending the time before the steel loses its integrity and the building collapses, thereby allowing additional time for safe evacuation.

During a fire situation, a steel structure will heat up, the rate of heating  
15 depending on the specific dimensions of the steel sections used in the structure. The rate of heating is dependent on the  $H_p/A$  value of the section, where  $H_p$  is the perimeter of the steel when viewed in cross-section, and  $A$  is the cross-sectional area.

A steel section with a large perimeter ( $H_p$ ) will receive more heat than one  
20 with a smaller perimeter. On the other hand, the greater the cross-sectional area ( $A$ ), the more heat the steel section can absorb. Thus, a large thin steel section having a high  $H_p/A$  value will heat up more quickly than a small thick section having a lower  $H_p/A$  value.

The thickness of the coating that is applied depends on the Hp/A value of the steel, its configuration, and the level of fire protection required. The latter is typically specified from 30 minutes to 120 minutes, this being the time taken for the steel to reach its critical failure temperature (550°C) under standard test  
5 conditions.

Typically the dry film thickness of intumescent coating applied varies from 250µm to several millimetres, depending on the level of fire protection required. With solvent based or water based prior art intumescent coatings, the higher dry film thicknesses can only be achieved by the application of multiple coats.

10 Prior art intumescent coatings designed for cellulosic fire protection tend to be based on high molecular weight thermoplastic resins based on acrylate, methacrylate and/or vinyl chemistry and require a high proportion of organic solvent or water to facilitate application to the substrate to be fire protected. This leads to slow and often protracted drying times, especially when high wet film  
15 thicknesses are applied (up to 2mm per coat), since the rate of drying is dependent on the evaporation of the carrier solvent. Increasingly stringent legislation concerning organic solvent emissions has meant greater use of water based products but slow drying remains a problem, particularly when the relative humidity is high.

20 Where multiple coats are required the problems of slow drying are exacerbated, particularly with solvent based coatings, where solvent from subsequent coats can strike back into the previous coats.

The use of ovens or near infra-red heaters can reduce the drying times, though these are expensive to operate, and due to the thermoplastic nature of the coatings, cooling is necessary prior to handling in order to prevent damage. Cooling of thick heavy steel sections, can take a relatively long time.

5       Traditionally, intumescent coatings were applied on-site to steel structures during the construction phase of the building. More recently in-shop application has become more common practice, as better control of application conditions is possible. Slow drying means that throughput is compromised, as coated steel sections cannot be moved until they are hard enough to resist damage.

10       The present invention seeks to provide a relatively quick-drying intumescent coating formulation.

According to the present invention there is provided a liquid intumescent coating composition comprising a resin system comprising at least one polymeric component, one or more ethylenically unsaturated monomeric components, and at  
15       least one intumescent ingredient curable to a solid state on initiation by organic peroxide.

The invention provides a quick-drying coating composition in that the organic peroxide initiates the conversion of the intumescent ingredients into a solid state via a free-radical reaction. No organic solvent or water is necessary to  
20       reduce the viscosity in order to facilitate application of the coating as the reactive monomer facilitates this.

The coating has particular, but not exclusive application in the coating of steel structures to provide protection against fire by forming an intumescent and

insulating char. These coatings are suitable for both on-site and in-shop application.

Steel sections and other materials that are coated with such an intumescent coating composition harden much more rapidly than prior art materials, since the  
5 drying time is dependent on the relatively rapid free-radical chemical reactions rather than on complete evaporation of volatile components. Drying times are reduced from 24 hours or longer, to around 30 minutes, (or even less with the incorporation of additional accelerator). This provides significant benefits to in-shop applicators, and enables a continuous process of application, drying and  
10 removal of steel sections from the application area.

Another benefit is that thick films can be applied in a single coat application, further reducing drying times compared with multiple coats of prior art intumescent coatings.

The present invention utilises free radical cure and comprises at least one  
15 solid thermoplastic polymeric resin component, combined with lower molecular weight liquid monomeric (or oligomeric components) containing ethylenically unsaturated double bonds. Preferably the ethylenically unsaturated double bonds are present as alpha-beta ethylenically unsaturated carboxylate ester groups such as methacrylate or acrylate groups.

20 The solid thermoplastic polymer is preferably a (meth)acrylic resin, either as a homopolymer, copolymer or terpolymer. The polymeric component ideally comprises a meth(acrylate) copolymer. This may be produced from the polymerisation of one or more methacrylate and acrylate monomers, such as any of the following:- methyl methacrylate, ethyl methacrylate, n-butyl methacrylate,

isobutyl methacrylate, t-butyl methacrylate, 2-hydroxy ethyl methacrylate, 2-hydroxy propyl methacrylate and the corresponding acrylates. Co-reactants may include styrene and vinyl toluene. The preferred solid thermoplastic polymer is a copolymer of butyl methacrylate and methyl methacrylate.

5 Other options for the solid thermoplastic polymeric resin component include homopolymers, copolymers or terpolymers derived from vinyl monomers such as any of the following:- styrene, vinyl toluene, vinyl chloride, vinyl acetate, vinylidene chloride and vinyl versatate esters. Co-reactants may include dienes such as butadiene.

10 The solid thermoplastic resin preferably constitutes from 20% to 50% by weight of the resin components of the coating composition.

The liquid monomeric components preferably contain methacrylate functionality, and most preferably are methacrylic acid esters. Additionally the monomeric components should preferably be monofunctional, in order that the  
15 resultant polymer produced on reaction with an organic peroxide is thermoplastic and thus melts and flows prior to temperatures at which the intumescent ingredients react.

Examples of suitable methacrylic acid esters include any of the following either alone or in combination:- methyl methacrylate, ethyl methacrylate, n-butyl  
20 methacrylate, isobutyl methacrylate and t-butyl methacrylate. Methyl methacrylate is most preferred due to its ability to produce low viscosity solutions of the solid thermoplastic resin component and its high glass transition temperature. However, its high volatility and characteristic odour, may for certain applications mean that alternative methacrylic acid esters may be preferable.



The liquid monomeric components preferably constitute from 50% to 80% by weight of the resin components of the coating composition.

Proprietary resin solutions containing both the aforementioned solid resin and liquid monomers may also contain oligimeric components.

- 5        The total resin components ideally constitute from 20% to 60% of the coating composition. More preferably the total resin components constitute from 30% to 50% of the coating composition.

One or more organic peroxides are added prior to use to convert the liquid coating to a solid state on the substrate. These are required to initiate the free-radical curing mechanism necessary to convert the monomeric (and oligomeric) components to a solid state. Suitable classes of organic peroxides include any of the following either alone or in combination:- diacyl peroxides, ketone peroxides, peroxyesters, dialkyl peroxides, hydroperoxides and peroxyketals. Diacyl peroxides are preferred, the most preferred peroxide in this class being dibenzoyl peroxide, which may be used in its solid granular form or as a paste with plasticiser.

10  
15

The organic peroxide content is determined by the total resin content, and preferably constitutes from 0.5% to 5% of the total resin content. More preferably the organic peroxide constitutes from 1% to 3% of the total resin content.

- 20        Where dibenzoyl peroxide is used as an initiator, a tertiary amine is preferably added to the coating compositions to accelerate the rate of cure, thus allowing the pot-life and curing time to be optimised. Aromatic tertiary amines are preferred, suitable examples include any of the following:- N,N-dimethylaniline and N,N-dialkyl-p-toluidine compounds such as N,N-dimethyl-p-toluidine and

N,methyl-n-hydroxyethyl-p-toluidine. The most preferred aromatic tertiary amine accelerator is N,N-dimethyl-p-toluidine.

The tertiary amine content is determined by the total resin content, and ideally constitutes from 0.1% to 4% of the total resin content. More preferably the  
5 tertiary amine constitutes from 1% to 3% of the total resin content.

The intumescent coating compositions of the present invention also contain specific ingredients that react together under the influence of heat from a fire, to form a protective insulating foam or char. These ingredients should be of low solubility to ensure that the coating has an acceptable level of durability, and  
10 maintains its integrity throughout the service life of the coating. Additionally, the intumescent ingredients used should have a sufficiently small particle size, in order to obtain satisfactory dispersion in the resin components, and thus allow application by spray application methods giving high rates of transfer of the coating to the substrate.

15 The intumescent ingredients preferably consist of three components, an acid source, a carbon source and a spumific or gas source. Preferably a nucleating agent should be present and optionally additives may be added to aid char formation and strengthen the char.

Under the influence of heat (between 100°C and 200°C) the resin  
20 components melt and begin to flow. As the temperature increases (>200°C) the acid source, usually by decomposition, produces copious amounts of acid which can react with other constituents in the coating. If the acid source is ammonium polyphosphate, polyphosphoric acids are released which can react with polyhydric

alcohols such as pentaerythritol (carbon source) to form polyphosphoric acid esters. The decomposition of these esters leads to the formation of carbon compounds, which together with a blowing agent such as melamine, give rise to a carbon foam or char.

5        The intumescent coating compositions of the present invention ideally contain at least one acid source, examples of which include ammonium polyphosphate, melamine phosphate, magnesium sulphate and boric acid. The preferred acid source is ammonium polyphosphate.

10        Ammonium polyphosphate can vary in molecular weight (chain length), the lower the molecular weight, the higher the solubility. By having very high molecular weight and a cross-linked structure it is possible to have very low water solubility, though higher thermal stability is observed. Coating ammonium polyphosphate with silane, melamine or melamine formaldehyde is beneficial in further reducing solubility and can also lead to higher loadings due to a reduction  
15        in resin absorbing properties. The use of coated ammonium polyphosphate is preferred, and ammonium polyphosphate coated with melamine formaldehyde is most preferred.

      The acid source preferably constitutes from 35% to 65% by weight of the intumescent ingredients content of the coating composition.

20        The intumescent coating compositions of the present invention ideally contain at least one carbon source, examples of which include polyhydric alcohols such as pentaerythritol, and dipentaerythritol. Starch and expandable graphite are other possible carbon sources. The preferred carbon sources are pentaerythritol and dipentaerythritol or a combination of the two.

The carbon source preferably constitutes from 10% to 40% by weight of the intumescent ingredients content of the coating composition.

The intumescent coating compositions of the present invention ideally contain at least one gas source, examples of which include any of:- melamine, 5 melamine phosphate, melamine borate, melamine formaldehyde, melamine cyanurate, tris-(hydroxyethyl) isocyanurate (THEIC), ammonium polyphosphate or chlorinated paraffin. The resin itself may be a gas source as it undergoes decomposition. The preferred gas source is melamine.

10 The gas source preferably constitutes from 5% to 35% by weight of the intumescent ingredients content of the coating composition.

Although not an essential ingredient in intumescent reactions, nucleating agents are a preferred ingredient since they promote sites for the intumescent char to form. The intumescent coating compositions of the present invention contain at least one nucleating agent, examples of which include titanium dioxide, silica, 15 silicates, mica and bentonite clay. A preferred nucleating agent is titanium dioxide which also provides opacity to the coating.

The nucleating agent preferably constitutes from 1% to 25% by weight of the intumescent ingredients content of the coating composition.

Further optional additives may be optionally included as part of the 20 intumescent ingredients to aid char formation and to strengthen the char and prevent char degradation.

The total intumescent ingredients ideally constitute from 40% to 80% of the total coating composition. More preferably the total intumescent ingredients constitute from 50% to 70% of the total coating composition.

In order that the intumescent coating compositions of the present invention can be applied at high film thickness in a single coat application it is preferred to modify the rheology of the coating by the incorporation of a thixotrope. Suitable thixotropic additives include organically modified inorganic clays such as bentonite clays, hectorite clays or attapulgite clays. Organic wax thixotropes based on castor oil are also suitable and are most preferred, due to their superior flow characteristics at high temperature.

The thixotropic additive preferably constitutes from 0.1% to 2% of the total coating composition. A more preferred level is from 0.5% to 1%.

The components of the intumescent coating compositions, with the exception of the organic peroxide initiator, are preferably blended together by the coating manufacturer using high speed dispersion equipment, whereby the solid intumescent ingredients are wetted out and dispersed in the resin components. Optional dispersion aids may be incorporated to facilitate this process.

The thickness of the coating is ideally at least 250 $\mu$ m.

Prior to application of the coating the organic peroxide is incorporated into the bulk of the liquid coating. This initiates the free radical reactions that will convert the liquid coating to its solid state. Typically, the initiated liquid coating will remain liquid and suitable for application for up to 30 minutes, though this can be modified by varying the quantities of initiator and accelerator in the formulation.

Suitable preferred methods of application of the aforesaid compositions include airless spray, brush, roller, trowel and dipping. Airless spray is most preferred. Airless spray pumps having a ratio of 45:1 or greater, and preferably

60:1 are suitable. A minimum air pressure of greater than 60 p.s.i. and preferably 80 p.s.i. is required, and the compositions are sprayed using a tip size ranging from 0.021 inch and 0.035 inch.

The coating compositions should be stored under cool conditions, and ideally application should only be carried out under such conditions. Where it is necessary to apply the coating compositions at higher temperatures, then modified formulations that have been adjusted for initiator or accelerator should be used.

The coating compositions of the present invention can be applied in liquid form to steel sections up to several metres in length with a gauge thickness typically ranging from 5mm to 30mm or greater. Depending on the  $H_p/A$  of the steel section coating can be applied at the required thickness to achieve 120 minutes fire protection.

Steel sections requiring fire protection are normally blast cleaned prior to the application of an intumescent coating to remove millscale and other deposits that may lead to premature failure of the intumescent coating, either on prolonged atmospheric exposure or during a fire situation. In order to prevent deterioration of the blast cleaned surface, particularly where there is a delay in applying the intumescent coating, it is normal practice to apply a primer coating. This is often the case when the intumescent coating is applied on site.

Examples of suitable primers are coatings based on epoxy, modified epoxy (such as modified with polyvinyl butyral), polyurethane, acrylic, vinyl and chlorinated rubber. Primers based on epoxy are preferred.

The thickness of the primer is ideally in the range from 15 microns to 250 microns. Preferably the thickness should be in the range from 25 microns to 100 microns.

A topcoat may be applied to the cured intumescent coatings of the present invention, particularly to provide colour to exposed steelwork. A topcoat if correctly formulated will also enhance the durability of the intumescent coating compositions. A clear sealer may also be suitable.

Examples of suitable topcoats are coatings based on epoxy, polyurethane, alkyd, acrylic, vinyl and chlorinated rubber. Topcoats based on polyurethane are preferred.

The thickness of the topcoat can vary from 15 microns to 250 microns. Preferably the thickness should be in the range from 25 microns to 75 microns, as too high a thickness of topcoat may inhibit the intumescent reactions.

In order that the present invention may be more readily understood, a specific example thereof is set out below.

<u>Example formulation:</u>	<u>Parts by weight.</u>
Titanium dioxide	9.80
Ammonium polyphosphate	29.00
Pentaerythritol	8.20
Melamine	10.50
Castor oil based thixotrope	0.70
(Meth)acrylic polymer (40% in methyl methacrylate)	31.60
Methyl methacrylate monomer	<u>10.20</u>
	100.00

The above example formulation was applied to a ½ metre steel I-section having a web length of 203mm, a flange length of 203mm and a weight of 52kg per metre ( $H_p/A = 180$ ). The mean dry film thickness was measured at 1625 microns, after being allowed to dry for 5 days.

- 5        The steel section was fire-tested in a  $1m^3$  furnace according to BS476 Part 20, 1987 at Warrington Fire Research Centre. The time taken for the steel section to reach the Critical Failure Temperature ( $550^\circ C$ ) was 63 minutes.

It is to be understood that the above described embodiment is by way of example only. Many modifications and variations are possible.